From: Crawford, Beth
To: Moore, Gary
Cc: Cassidey, LeRoy
Subject: FW: Detection-limit issues/possible solutions Houston Tanks
Date: Wednesday, October 22, 2014 9:44:36 AM
Attachments: removed.txt

Gary,

Please see Guy's response on the matrix interference question we had yesterday. Let me know if you have any questions.

Thanks,

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From: Gallello, Guy
Sent: Wednesday, October 22, 2014 10:32 AM
To: Crawford, Beth
Cc: Cassidey, LeRoy
Subject: Detection-limit issues/possible solutions Houston Tanks

Beth,

As we discussed yesterday, the reporting limit and dilution problems the lab is seeing with these very difficult samples is understandable and they appear to have exhausted the capabilities of the methods used to acquire "usable" data. There are however alternative means of further increasing sensitivity and thereby reducing detection/reporting limits that will hopefully eliminate what appear to be "false" D-codes due to the inability to analyze these samples at reporting/detection limits adequate for characteristic determination for select analytes.

Since the methods in SW-846 are considered to be guidance and there is allowed flexibility, via the Methods Innovation Rule, to the project/laboratory to achieve usable data, the project and laboratory are permitted to work together towards a common goal of obtaining usable information and to modify the methods as necessary. The very difficult matrices, including even for TCLP, have already been shown to be resistive to typical clean-up methods and there appears to be little that can be accomplished at the sample preparation end to improve sensitivity and lower reporting/detection-limits. There are however, ways to enhance the detectability of the false-coded analytes at the instrumental analysis end of the methods, primarily by increasing the analytical selectivity and therefore sensitivity, the best one being Selective Ion Monitoring (SIM) GC/MS. The technique can be used either to reanalyze extracts still in hold to provide a lower detection limit for selected analytes or it can also be utilized as an alternative to other and even non-GC/MS analysis.

Since the technique forces the detector to stay focused on specific and characteristic ions and not to scan a large mass range it effectively reduces interference from constituents within the matrix and increases sensitivity by as much as 10-fold depending upon the analyte(s) of interest. Such an increase in sensitivity would provide data for the two samples we discussed at reporting/detection limits well below the regulatory limits and thereby serve to nullify the false D-codes the current data leads to.

Of course, the laboratory would have to be able and willing to perform the proposed analysis, and it would justifiably so, expect to be compensated accordingly. Assuming that they can be persuaded to assist, here is what I would recommend for the two samples we discussed yesterday.

FT-1004 Aq layer

TCLP data for Hexachlorobenzene (HCB) and 2,4-dinitrotoluene (2,4-DNT) is BRL with MQL values above the regulatory limits, essentially D-coding the waste for both analytes. The TCLP SVOC extract was prepped on 9/8 and is therefore out of analysis hold-time (10/18). The sample was collected on 8/29 so a new sample will need to be collected and submitted to the laboratory.

It should be TCLP extracted (if required) and then extracted for targeted analysis of HCB and 2,4-DNT only via SW-8270D with the detector operating in the SIM mode. The laboratory can utilize the same internal standards (inject less into the extract) and at least one B/N surrogate. They can set-up the SIM parameters and calibrate to only target the necessary ISTD(s), analytes, and surrogate(s) needed to provide viable data.

FT-506

This sample is being false D-coded for several chlorinated pesticides (chlordane, heptachlor, and heptachlor epoxide) in the TCLP extract. The sample is now also out of hold so a new sample will need to be collected. Pesticides and even PCBs can also be analyzed using GC/MS-SIM and in fact EPA-680 was specifically set-up to do so and SW8270D lists the pesticides and PCBs as possible analytes. If the laboratory has exhausted the available extract clean-up methods (Florisil™ etc.) and not been able to reduce the matrix interference sufficient to allow for ECD detection limits sufficient to provide viable data with RLs below the regulatory limits, the GC/MS-SIM method can be used to target the analytes of interest in a specially designed and calibrated analysis. The characteristic ions for each of the analytes can even be found in SW-8270D as these analytes are listed as possible analytes for the method.

The above solutions could be applied to any other problem analytes/samples on a case by case basis. Alternatively, you may want to consult with the laboratory and see if they are currently running a trace detection version of SW8270D in the SIM mode routinely. If so, you could request that further difficult matrix samples be analyzed for TCLP/SVOCs and pesticides using it.

Hope this clears things up and helps and I would be willing to assist further in any way,

Guy

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********** ATTACHMENT REMOVED
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